

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1091a

Oxygen in Stainless Steel (AISI 431)

This Standard Reference Material (SRM) is intended for use in the evaluation of methods and the calibration of equipment used in the determination of oxygen in steels or ferrous materials. SRM 1091a is in the form of a stainless steel rod, 0.79 cm diameter and 10.2 cm long (5/16 in. diameter and 4 in. long).

The certification of this SRM is based upon results from two independent techniques, inert gas fusion infrared thermal conductivity and 14 MeV neutron activation analysis. A minimum of 1 g of the material should be used for any analytical determination.

Oxygen concentration - - - - - 132.2 ± 3.1^a $\mu\text{g/g}$

^aThe above listed uncertainty represents \pm two standard deviations of the certified value and includes the observed variability between samples.

Procedure for Use

In order to properly use this SRM the user is cautioned to carefully carry out the following procedure:

Cut the sample to an appropriate size using a silicon carbide, water-cooled, cut-off wheel or by other means that will avoid overheating. Avoid oxide cutting or oxide abrading materials. File all surfaces of the sample to remove oxides or other impurities, rinse in acetone, air-dry, and weigh. Be sure that all pores in the surface of the sample are free of acetone. Do not touch the sample with fingers during and following the final stages of cleaning. Store the prepared samples in a desiccator. If they are not analyzed within four hours of preparation, repeat the preparation procedure beginning with the filing step prior to analysis.

The analytical work for homogeneity testing and certification was performed in the Inorganic Analytical Research Division at NBS by B.I. Diamondstone and R.C. Gauer.

Cooperative analyses for certification were performed at the United States Steel Corporation by M.J. Nardozzi and M. Kotyk.

The statistical analysis was performed in the National Measurement Laboratory by R.C. Paule.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T.E. Gills.

Gaithersburg, MD 20899
November 1, 1985

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

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The octaphenylcyclotetrasiloxane was prepared by the Silicone Products Department of the General Electric Company and obtained through the Eastman Kodak Company of Rochester, N.Y. Chemical analyses were conducted by B.B. Bendigo, activation analyses by S.S. Nargolwalla and J. Suddueth, and spectrochemical analyses by V.C. Stewart.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of P.D. LaFleur.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Standard Reference Materials Program by T.W. Mears.

DIRECTIONS FOR PREPARING LUBRICATION-OIL SOLUTIONS OF OCTAPHENYLCYCLOTETRASILOXANE

Transfer approximately 0.2 g of this compound from the bottle to a small beaker and dry over fresh phosphorus pentoxide in a desiccator for 2 hours. (Tightly close the bottle containing the remainder of the compound.) Quickly and accurately transfer 0.141 g of this compound to a weighed 200-ml flask. (This weight of compound is equivalent to 20 mg of silicon.) Add 4 g of xylene and heat the flask on a hot plate, with swirling and without charring, until the solid dissolves. In a separate flask, heat 95 g of lubricating oil to 70 °C, and carefully pour this hot oil into the silicon solution. Allow the solution to cool to room temperature and add enough cool lubricating oil to bring the total weight of the contents of the flask to 100 ± 0.5 g. Reheat the flask immediately to 75 °C under reflux and keep the solution under reflux at this temperature during use. The concentration of silicon in this solution is 200 ppm.

Preparation, Testing and Analysis

The base material for SRM 1091a is a stainless steel (AISI 431) alloy that was obtained from the Jones and Laughlin Steel Corporation in the form of centerless ground rods 0.79 cm in diameter and 365.8 cm long (5/16 in. diameter and 12 feet long) and prepared from a single heat. This high alloy steel contains a high percentage of chromium which could conceivably be a source of some difficulties in oxygen determination. The material has a nominal composition as follows: C= 0.15%, Ni= 2.2%, Cr= 15.8%, V= 0.04%, N= 0.09%. Homogeneity testing was performed on samples from all rods of this SRM lot.

SUPPLEMENTARY INFORMATION

Although not certified, the nitrogen content of this SRM was determined by the analytical methods shown below:

<u>Methods of Analysis</u>	<u>Nitrogen (ppm by weight)</u>		
	<u>\bar{x}</u>	<u>s</u>	<u>n</u>
Semi-micro Kjeldahl	878	13	60
Inert gas fusion thermal conductivity	874	4	14

\bar{x} = mean nitrogen value; s = standard deviation of a single determination; n = number of independent determinations